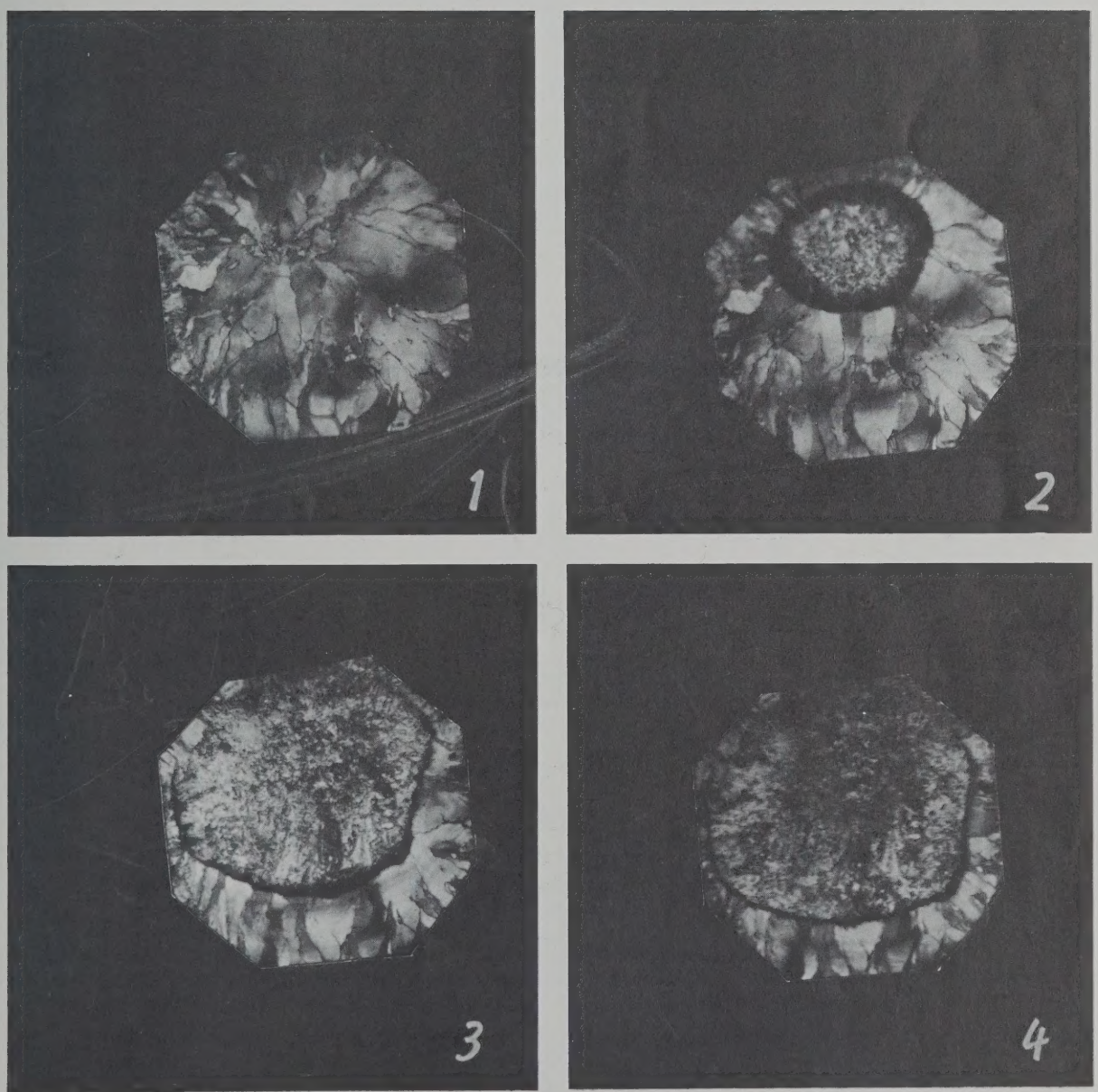
U.S. Department of Commerce

NATIONAL BUREAU OF STANDARDS

Technical News

BULLETIN



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U.S. DEPARTMENT OF COMMERCE — LUTHER H. HODGES, *Secretary*

NATIONAL BUREAU OF STANDARDS — A. V. ASTIN, *Director*

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COVER: A new technique developed at the National Bureau of Standards permits direct visual observation of the behavior of materials at extremely high pressures. On the cover is a series of four microphotographs, taken under polarized light, showing pressure effect on potassium nitrate crystals (× 160). As pressure is increased up to 2,900 bars, a growing crystal front that has the appearance of a liquid interface is produced. Under the microscope, the movement of the individual crystals can be observed as they grow. (See p. 112.)

Electron Resonance Magnetometer

A MAGNETOMETER [1] capable of measuring alternating magnetic fields of 2800 to 4300 gauss to an accuracy within 0.1 percent has been developed by O. E. Spokas and M. Danos of the high energy radiation laboratory. This device uses the technique of electron spin resonance (ESR) to determine the field strength. As the ESR probe is constructed entirely of dielectric materials, no distortions are introduced into the field being measured. The Atomic Energy Commission sponsored work on the magnetometer, which has been used to measure the magnetic fields within the NBS betatron and synchrotron.

Electron spin resonance, a technique widely used in investigating the structure of molecules and free radicals, can be used equally well for determining the strength of magnetic fields. In essence, ESR depends upon the fact that, in an applied magnetic field, materials having unpaired electrons will resonate when the proper radiofrequency is applied. The resonant frequency, which can be determined in a number of ways, depends upon the material used and the strength of the magnetic field.

The ESR technique was selected because the detecting element could be made small enough to minimize the effects of field inhomogeneities. In addition, the sensing material has a sufficiently short relaxation time to respond to the rates of field change present in the NBS accelerators (about 5 gauss/μsec). Nuclear magnetic resonance technique is unsuitable for this application due to the bulk of the sensing material and its long relaxation time.

In this magnetometer 1,1-diphenyl-2-picryl hydrazyl (DPPH) is used as the detecting material. The resonant frequency depends on the relation

$$F = (2.804 \pm 0.001)\ H,$$

where F is the resonant frequency in megacycles per second and H the field in gauss. Using an X-band klystron (8 to 12 Gc/s), field strengths in the range 3000 to 4000 gauss can be determined. The use of higher frequencies would, of course, permit the determination of higher fluxes.

The ESR probe consists of a section of dielectric waveguide, with the DPPH field sensor on one end and a matching section on the other. The dielectric guide is fabricated of ceramic-filled polystyrene, a commercial product available in a range of dielectric constants. The probe used in most of the measurements was 0.6 m long, with a cross section trimmed to permit insertion into X-band waveguide. A plastic sack containing 5 mg of DPPH powder is inserted in a depression in the face of the sensing end of the probe. A titanium dioxide plate is cemented to the face to intensify the spin resonance signal, and to retain the sensing material. A second pouch of DPPH placed between the first TiO_2 plate and a second similar plate is required on probes that are to be used for field gradient measurements (see illustration). The matching section, which consists of a polyethylene quarter wave matching transformer cemented to the probe, a waveguide horn, and a sliding stub tuner, provides a smooth transition between the dielectric guide and the air-filled guide. Probe length, cross section, dielectric constant, and matching technique may be altered to suit various requirements. Under certain conditions as little as one granule of DPPH (0.1 mg) will generate a useful signal.

A microwave bridge technique is used to determine the resonance condition of the DPPH sample. This is done by dividing the continuous wave klystron output between an adjustable termination and the probe. Resonance within the probe unbalances the bridge, and causes an RF pulse to travel down the waveguide. After demodulation and amplification, this pulse is displayed on an oscilloscope, together with a time reference pulse. A tunable X-band klystron is used to provide the necessary radiofrequency, and conventional components are used throughout the waveguide system. A more complex FM system was also developed which generates well-defined resonance signals in slowly changing fields. However, the simpler CW system was used to make most of the measurements. Field measurements are made by comparing the ESR signal with

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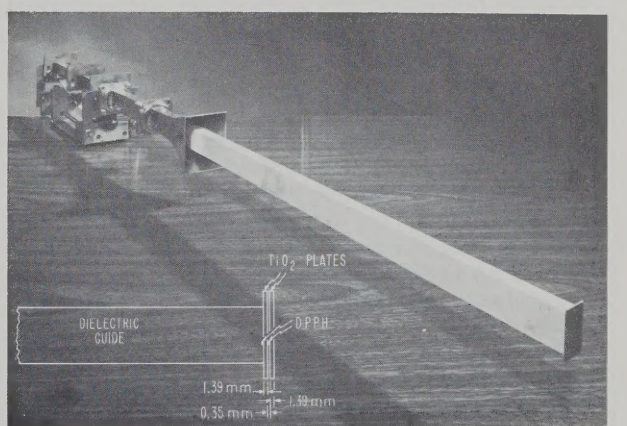
Electron spin resonance magnetometer for the determination of time and space varying magnetic fields of 2800 to 4300 gauss. The sensor consists of 5 mg of 1,1-diphenyl-2-picryl hydrazyl imbedded in the front surface of the probe and covered with a titanium dioxide plate which enhances the resonance effect. The probe is made of ceramic-filled polystyrene, and thus introduces no distortions in the field being measured. Field-gradient measurements can be made by sandwiching a second sensor between the existing and another titanium dioxide plate (*see inset, lower left*).



a suitable time reference signal derived directly from the magnetic field.

The magnetometer has been used to determine the effects of machine warmup on the field at points within the betatron donut, and to obtain a plot of the field in a horizontal plane along the injector axis. It indicated the existence of a 0.2 percent nonlinearity in the betatron energy-control apparatus. Spatial field gradients in the betatron have been determined to within 10 percent using the double sample probe. This figure can easily be improved upon. It has also been used to determine the characteristics of new energy control equipment for the synchrotron. Some of these measurements indicate that an accuracy of ± 1 gauss in the magnetometer reading is quite feasible.

The physical size and shape of the probe may be altered to permit use of the instrument in areas not easily reached by the probe shown in the illustration. Time and space varying magnetic fields may be investigated in electrical machinery and equipment with safety due to the dielectric nature of the probe.

Dielectric strip-guide techniques may permit investigation of magnetic field distribution within the gap of large electrical rotating machinery. The magnetometer may also be used as a source of a precision trigger or marker generated at some predetermined field strength.

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[1] An electron resonance magnetometer for alternating magnetic fields, *O. E. Spokas and M. Danos, Rev. Sci. Instr.* **33,** *613 (June 1962).*

Eighth Scintillation and Semiconductor Counter Symposium

OVER 700 scientists from North America and Europe attended the 8th Scintillation and Semiconductor Counter Symposium, in Washington, D.C., from March 1 to March 3, 1962. The Symposium consisted of 6 sessions, in which over 50 technical papers were presented, and a dinner session addressed by Dr. L. J. Hayworth, Commissioner of the U.S. Atomic Energy Commission. Held at 2-year intervals, this meeting regularly attracts leaders in the field of radiation detection and measurement. It is sponsored jointly by the National Bureau of Standards, the Atomic Energy Commission, the Institute of Radio Engineers, and the American Institute of Electrical Engineers.

The first half-day session was concerned with developments in scintillators and with understanding the response of scintillators. It was conducted under the chairmanship of Prof. Robert Hofstadter of Stanford University, who recently shared the Nobel Prize in physics. The current status of photomultipliers and image tubes was reviewed in the second session which, with Louis Costrell of NBS as chairman, was comprised of papers describing recent developments in the United States, Britain, and France.

The session on counter systems and applications, of which W. A. Higinbotham of Brookhaven National Laboratory was chairman, dealt with instrumentation, high-speed techniques, and particle discrimination techniques. A review paper on particle type discrimination by phosphor decay was presented by R. B. Owen of the British Atomic Energy Research Establishment, who did pioneer work on this subject. Highlights of the semiconductor radiation detectors session, of which C. J. Borkowski of the Oak Ridge National Laboratory was chairman, were a review paper by J. W. Mayer of Hughes Aircraft Company and a comprehensive paper on the use of semiconductor detectors in nuclear physics, delivered by Prof. D. A. Bromley of Yale University. Developments of the last 2 years that have pushed these detectors to the forefront in heavy particle physics were described at this session.

The session on track imaging and spark chambers, conducted with G. T. Reynolds of Princeton University as chairman, featured a review of the status of the scintillation chamber by M. L. Perl of the University of Michigan. This was followed by a comprehensive review of spark chambers, currently receiving much attention in the high-energy accelerator field, presented by J. W. Cronin of Princeton University. The final session, on space applications, included papers on both scintillation counter and semiconductor detector spectrometry, presented under the chairmanship of F. B. McDonald of the Goddard Space Flight Center. The proceedings of the Symposium were published in the June 1962 issue of the Transactions of the Professional Group on Nuclear Science of the IRE.

The symposium series, of which this is the eighth, began in July 1948 with a meeting held at the University of Rochester under the sponsorship of the University, the Atomic Energy Commission, and the Office of Naval Research. This pioneer meeting was concerned with both scintillation counters and crystal counters. The scintillators described included naphthalene, anthracene, and sodium iodide. The work reported on crystal counters was concerned primarily with silver chloride, silver bromide, and diamonds. The second meeting, sponsored by the Oak Ridge National Laboratory and held at Oak Ridge, Tennessee, in June 1949, was confined to scintillation counting.

The 1952 meeting was held at Washington, D.C., where it has been held under the present sponsorship every 2 years since. In 1960 crystal counters (semiconductor detectors) again occupied the attention of the symposium, resulting in change of its name in 1962 to the Scintillation and Semiconductor Counter Symposium. The next symposium in the series is scheduled for 1964 at Washington, D.C.

Unique Laboratory Designed for Radiation Physics Experiments

THE NEW NBS Radiation Physics Laboratory at Gaithersburg will house, in one building, a variety of facilities for radiation research. The accelerators and related equipment in the laboratory will be used to produce, measure, and utilize ionizing radiations ranging in energy from 5,000 ev to 200 Mev. These facilities will be used in the development of standards and equipment for the measurement of high-energy radiation, and the study of the interactions of high-energy radiation with matter and its elementary constituents.

The Radiation Physics Division does three different types of work: research with high-energy accelerators and sources, laboratory research on radioactive materials, and electronic and theoretical investigation involving little or no contact with radiation. It appeared at first that it would be necessary to separate these kinds of work in individual buildings. However, housing the entire division in a single building would enable scientists to work in more than one of the subject areas and simplify communications among the scientific staff.

(*Continued next page*)

Architects' rendering of the administration building at the new NBS laboratories in Gaithersburg, Md.

Contract Signed for Second Construction Phase of NBS-Gaithersburg Facilities

On June 19, 1962, Secretary of Commerce Luther H. Hodges announced the signing of a $25,328,000 contract for the second construction phase of the Bureau's new laboratories in Gaithersburg, Md. The contract, which was awarded by the General Services Administration to the Blake Construction Company, Inc., of Washington D.C., includes construction of five buildings as well as site work, roadwork, and outside utilities on the north portion of the site. The buildings to be constructed include the Radiation Physics Laboratory, the Administration Building, and three other buildings for supporting services, including the scientific instrument shop. These buildings will be completed in 1965.

The Engineering Mechanics Laboratory and the Power Plant, which make up what has been known as Phase I of the construction program, are now under construction. The first two phases thus include two major special-purpose laboratories and the supporting service buildings necessary for the entire facility. Still to be contracted for are seven large research laboratory buildings. These general-purpose laboratories will house approximately 1,500 of the NBS Washington staff of 2,700 employees.

The Administration Building includes facilities for scientific symposia, meetings of professional societies, and NBS staff meetings. There will be 2 auditoriums, one seating 750 and the other seating 300. In addition, there will be lecture rooms for 100 and conference rooms for smaller groups, a completely equipped still and motion picture laboratory, and an NBS museum with instruments and equipment that have played significant and historic roles in the science of measurement. The NBS technical library with shelf space for 100,000 volumes in open stack areas will house extensive material in all of the physical sciences. A number of services which are scattered in various locations on the present Bureau grounds will be centralized in the Administration Building. These include the NBS Graduate School, a high-speed computer, and emergency medical care facilities.

The first floor and basement of the building cover a U-shaped area, 375 by 432 ft, with wings for the library and the large auditorium attached. A section 375 by 154 ft is the base of the 11-story office tower. The building will be connected with the seven general purpose laboratory buildings by glass-enclosed corridors.

Since this 11-story building will be the highest on the site, a penthouse will be built on the roof for the use of scientists doing experiments which require a clear line of sight from an elevation above the surrounding buildings.

It is planned to expand the building when necessary by additions to the library and cafeteria wings and by adding a second tower, probably seven stories high, for additional offices.

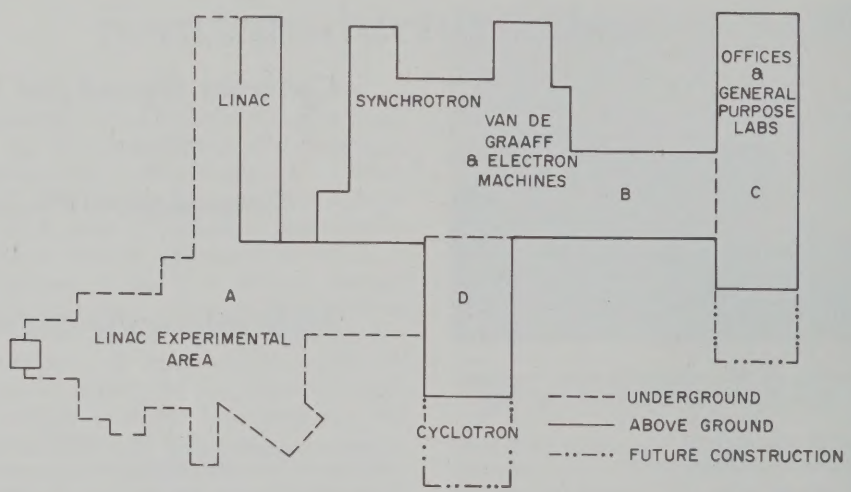
The new laboratories are being built on a 550-acre site approximately 20 miles northwest of Washington, D.C. Plans now call for 20 major buildings with a total assignable space of 1,200,000 sq ft. Total cost of the new facilities is estimated at $104 million. Design of the Gaithersburg laboratories is by the architectural firm of Voorhees, Walker, Smith, Smith, and Haines of New York City.

The building, as finally designed, will make it possible to carry on the various programs without radiation interference among the laboratories.

High-level radiation areas have been removed as far as possible from the low-level radiation areas. The most powerful NBS accelerator, the new Linac (linear accelerator), is placed in a separate wing, effectively three levels below ground at one end of the building, and at a distance of 400 ft from the three-story wing where the research requiring very low levels of background radiation is conducted. Near the linear accelerator are located the synchrotron, the Van de Graaff accelerator, and various X-ray machines. One other high-energy machine, a variable-energy positive ion accelerator, is planned as a future addition.

One other important criterion in the building design was the provision of radiation protection. In the design of areas housing high-intensity sources of radiation, the natural inexpensive shielding of earth fills was utilized. Where it is necessary to provide shielding between immediately adjacent areas, very thick concrete walls are used. Elaborate interlock mechanisms have been provided to prevent entrance into areas of high-level radiation. The entire system of stairs and passages has been designed on a labyrinthine principle to afford complete shielding integrity.

The sketch above shows the layout of the laboratory. It will have approximately 170,000 gross sq ft with about 71,000 sq ft of assignable space. More than 65 percent of the building will be underground. Above ground will be the three stories of wing C, where investigations involving little or no contact with radiation will be done.



Linac Area

The Linac is an electron accelerator of fairly recent design. This design was specially chosen by the Bureau staff to meet its needs; it has been contracted for and is now being built. When it is put into operation, it will produce one of the world's most intense high-energy electron beams, with energies continuously variable from 10 to more than 100 Mev and power outputs greater than 40 kw—a power output about 100,000 times that now obtained at the Bureau in the same energy range. The increase in intensity will make it possible for the Bureau to enter new areas of nuclear and atomic physics; and to establish standards, evolve measuring techniques, and determine shielding requirements for the high dose rates now being employed by industry to sterilize pharmaceuticals, to preserve food, to polymerize plastics, vulcanize rubber, etc. The 10 billion rad/hr dose rate of the Linac, added to those available from the Bureau's existing sources, from cobalt, radium, X-rays, and other electron-beam sources, will make it possible to conduct valuable investigations into problems of dosimetry for most of the important ranges that are at present in use or which can be anticipated in the near future.

An accelerator with such a large power output requires elaborate shielding to insulate the danger areas from the working areas. Such shielding can most simply be obtained by burying the accelerator deep underground. The Linac wing descends 32 ft below the height of the first floor in the rest of the laboratory and occupies three basement levels. On the lowest level, that is, the second subbasement, the Linac itself is located in a narrow room 156 ft long at the western edge of the wing. The machine is essentially a 100-ft pipe through which electrons are accelerated on the crest of a traveling wave. Controlled from a magnet room located at the eastern end of the Linac chamber, the electron beam is directed into one of three measurement rooms that are each adjacent to the magnet room but with shielding between—an important factor, for it permits concurrent occupancy of the other two measurement rooms for setting up experiments.

Shielding in the measurement rooms is provided by 12-ft-thick concrete walls. The measurement rooms can be entered without going through the magnet room, so that measurement rooms not receiving the beam can be entered safely even though the beam is passing through the magnet room. Entrance to the measurement rooms is via interlocking concrete horizontal-plug shielding doors, each weighing 50 tons. Air conditioning on this level will be a low-velocity single-zone reheat system, using 100 percent outside air at all times.

Access to the rest of the laboratory, at both the first and second subbasements, is by corridors running north to join wing D. At the level of the first subbasement, where the rest of the laboratory begins, the Linac chamber, magnet room, and measurement rooms are already covered over. On the first subbasement level of wing A are the Linac cooling room and the Linac power room, located immediately above the Linac chamber itself. Along the corridor leading to wing D are the Linac control room, maintenance room, and various laboratories.

A different system of air-conditioning operates on this floor: a low-velocity multizone reheat system which is designed to absorb the greater part of the heat released by the motor generator equipment in the power room and cooling room.

Framing is of reinforced concrete, and in some places "high-density" concrete is to be used for walls and slabs in order to conserve space.

Synchrotron Area

The synchrotron accelerator is now in use at the Bureau's present site. It will be transferred to the new laboratory and placed in the southwest corner of wing B. Two floor levels will be required to provide sufficient clearance for the machine. The synchrotron room is located in the basement and continues up to the level of the first floor ceiling. Also on the basement floor are the control room, assembly room or setup laboratory, and darkroom for developing films. Framing throughout the wing is of reinforced concrete with thick walls and slabs for radiation protection. The machine itself is enclosed by a demountable concrete shielding wall 6 ft thick.

Area for Van de Graaff and High-Energy Electron Accelerators

Also located in wing B is another high-energy radiation area which includes the positive ion Van de Graaff accelerator and two high-energy electron accelerators.

Since both the Van de Graaff accelerator and the two electron accelerators require rooms with heavy shielding, they have been placed close together to form a single high-radiation area. The Van de Graaff is used in neutron experiments. The electron machines may be utilized in two ways: The electron beams may be made to strike a target and thus produce X-rays, or they may be passed into a gas for a detailed examination of their interaction with matter. The X-ray energies available from the sources in this area are similar to the ones used for treatment of cancer and for radiography. Here they will be used to investigate the efficiency of X-ray production, the shielding efficiency of various materials, and to conduct dosimetric studies. The electron beams provide radiation sources for dosimetric studies at dose rates similar to those provided by the Linac but with considerably smaller penetrations.

The main experiment room for the Van de Graaff is a 40-ft by 40-ft room enclosed by insulated aluminum panels. It projects from the west elevation of wing B. Deuterons or protons are ejected by the machine, and directed by magnets to various targets where the neutrons are produced. Protection of personnel from the moderate strength neutron sources in this room is provided by distance only. No special shielding is required and the neutrons are allowed to penetrate through the walls. The purpose of the thin walls is to insure that neutrons not involved in the experiments will pass through the walls and not be reflected back into the room where they might cause interference with the experiments in progress.

The room extends for three floor levels, starting at the first subbasement level and terminating at the first-floor level. The Van de Graff accelerator is one level higher, on the first floor. The magnet room, from which the positive ion beam of the Van de Graaff accelerator is controlled, is located in the basement directly below the Van de Graaff room. Immediately to the east of it are: the control room; a heavily protected area 16 ft by 16 ft with walls 3 ft thick for special shielded experiments; and a general experimental laboratory.

The most powerful of the high-energy machines in wing B is the 3-Mev machine located in a 988 sq ft room starting on the basement floor and extending up to the first-floor level. The actual radiation area, a large heavily shielded room containing a pit for investigating the details of electron interactions with gases, is on the subbasement level. A 1½-Mev machine is also located in wing B and rooms associated with it are designed on a similar principle. The machine room is located at basement level and continues up to the first floor, with the shielded radiation area immediately beneath on subbasement level. In addition, a 500-kv X-ray machine now in use at the Bureau will be located next to the 1½-Mev machine room, occupying both basement levels.

Air-conditioning throughout this high-radiation area will be by a high-velocity dual duct design, and temperature within individual spaces will be controlled by room thermostats. Outside air will be utilized whenever conditions permit.

Other Radiation Areas

Almost all the other laboratories and work areas requiring shielding are situated in the two basements of wings B and C, although a few are also located in the basement and first floor of wing D. Shielding walls for these spaces vary in thickness from 8 to 24 in.

Apart from the area devoted to the high-intensity radiation machines, assignable space throughout the subbasement floor is given over almost entirely to the activities concerned with radiological equipment. This work is mainly concerned with X- and gamma-ray research, standardization, and calibration; and with applications to equipment used for industrial and medical purposes. X-ray standards are available over a range 5 to 250 kv stabilized to provide very constant output. Gamma-ray calibration ranges employing cesium 137 and cobalt 60 provide for dose rates from a few milliroentgens per hour to several thousand roentgens per hour. Beam traps consisting of 3-ft diam by 6-ft-deep lead-lined pits improve the vertical ranges by reducing scattered radiation. Similar horizontal beam traps extend beyond the building into the earth for the horizontal beam ranges. Because the entire area is located below ground level, no radiation protection barriers are required for floors or outside walls. This simplifies construction and reduces the cost of the installation.

Future Expansion

Besides the addition for the variable-energy positive ion accelerator mentioned, plans for later expansion will probably include construction of neutron time-of-flight tubes adjacent to the Linac experimental area. The size and topography of the site make it possible to contruct four such tubes using the Linac as a neutron source in experiments to measure neutron cross sections and the energy spectra of photoneutrons. The route these tubes will take is undecided, but it is probable that they will run in a north-south direction.

Long-range expansion of existing laboratory areas has been allowed for in two places: in wing C to the east and in wing A—the Linac complex—to the west, and the footings have already been designed to carry the future columns that may be required.

Versatile Electron Energy Analyzer

for Low-Temperature Studies

A METHOD for investigating the characteristic energy losses of electrons in solids at low temperatures has been developed by E. M. Hörl and J. A. Suddeth [1] of the atomic physics laboratory. The heart of the method is a versatile electrostatic analyzer lens [2] which is used to separate electrons according to energy loss and scattering angle. Although intended primarily for investigating thin films of solidified gases at liquid helium and liquid hydrogen temperatures, the lens is suitable for the study of any substance that is obtainable as a thin film.

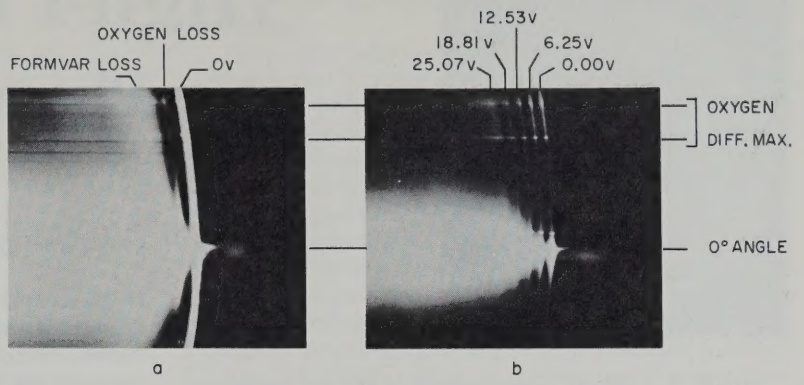
Characteristic electron energy losses in gases and solids have been studied for the past 30 yr, mainly at room temperature. However, theoretical considerations now indicate a possible temperature dependence of the energy-loss values in metals. The present method was developed in an effort to obtain experimental data that might resolve conflicting theoretical results regarding temperature influence.

In this method, thin layers of solid material are formed by condensation of gases on a thin foil maintained at low temperature. The electron beam passes first through the specimen film, then through the analyzer lens, and then onto a photographic plate. The electrostatic field of the lens causes the electrons to produce a pattern on the photographic plate from which data on electron energy losses can be obtained.

In the experimental arrangement used at the Bureau, a thin supporting foil (collodion, Formvar, or aluminum) is mounted over a hole in a target block of oxygen-free high-conductivity copper which is soldered to the bottom of a cylindrical container filled with liquid helium or liquid hydrogen. The temperature of the target block can be measured with a carbon resistance thermometer. The thin layers of condensed gas formed by condensation on the supporting foil are investigated by means of an electron beam of 30 kev energy. The target is completely surrounded by a radiation shield at a temperature of 20 °K. The shield has only three openings—for entrance and exit of the electron beam and for admission of the gas to be solidified on the substrate.

The analyzer lens possesses several useful features. Controls are provided outside the vacuum for positioning the slit-lens assembly with respect to the electron beam, positioning the slit with reference to the lens for optimizing resolution and dispersion, rotating the slit with respect to the lens, and for fine and coarse adjust-

Left: **Electron energy loss pattern obtained from a thin film of solid oxygen on a Formvar substrate at a temperature of 20 °K. The 9.5-ev energy loss of solid oxygen as well as the broad energy loss of the Formvar substrate at approximately 23 ev are indicated.** *Right:* **Calibration for energy loss pattern made by changing potential of electron gun filament with respect to center electrode of analyzer lens in approximately 6-v steps.**



ment of slit width. All controls are mounted on concentric shafts for ease of operation in a darkened room. "In-vacuum" friction is essentially eliminated by the use of phosphor bronze deformation members.[3] By shifting the complete lens assembly off the electron beam path, entire electron diffraction patterns of the samples can be observed and photographed before and after energy-loss measurements are made.

The electrostatic field of the lens causes two electrons having equal scattering angles but different energies to strike a photographic plate behind the lens at two different positions on a horizontal line. Electrons of equal energies but different scattering angles will hit the plate on a vertical line. The plate therefore becomes a two-dimensional plot of electron intensity (represented by the density of the plate) as a function of scattering angle (vertical coordinate) and energy loss (horizontal coordinate).

Patterns of this ideal type are obtained only with very small scattering angles when using the very central region of the lens. Larger scattering angles (up to 0.06 rad) cause bending of the vertical lines of equal electron energy. A calibration of the energy loss (horizontal coordinate) is made by changing the potential of the electron gun filament with respect to the center electrode of the analyzer lens in steps of approximately 6 v.

One of the electron energy-loss patterns obtained came from a thin film of solid oxygen deposited on a Formvar substrate at 20 °K. In addition to the energy loss for the Formvar substrate, an energy loss of 9.5 ev was observed for the oxygen.

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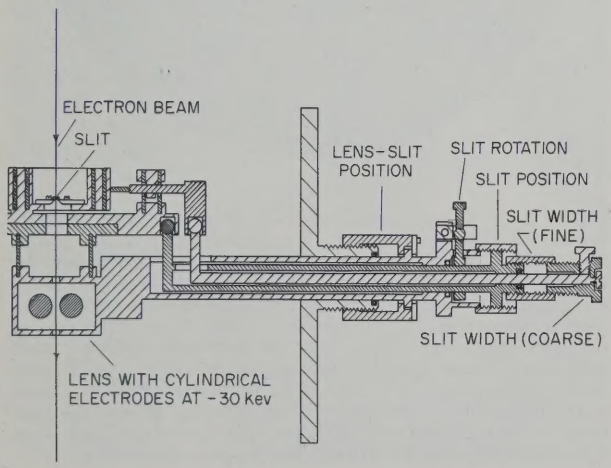
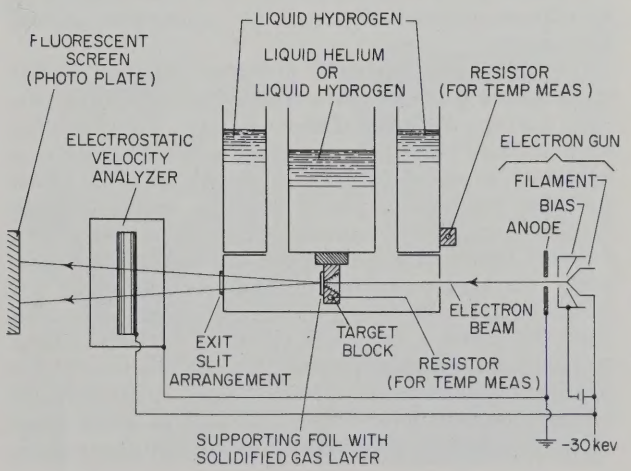
[1] *For further details, see* Characteristic energy loss measurements at low temperatures, by *E. M. Hörl and J. A. Suddeth, J. Appl. Phys.* **32**, *No. 12, 2521–2525 (1961).*

[2] *G. Mollenstadt, Optik* **5**, *499 (1949); ibid.* **9**, *473 (1952).*

[3] *R. V. Jones, J. Sci. Instr.* **28**, *38 (1951).*

Left: **Versatile electrostatic analyzer lens developed primarily for studying thin films of solidified gases. Figure shows a horizontal cross section through the lens and its control mechanism:** *Right:* **Vertical cross section of the experimental arrangement used to investigate characteristic energy losses of electrons in solids.**





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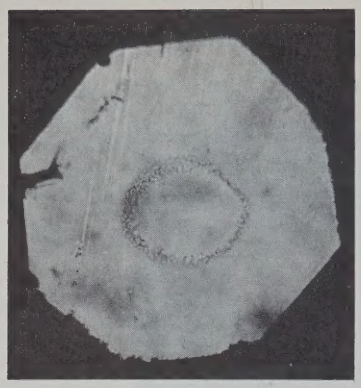


High-Pressure Optica

Phase Transitions Observ

DIRECT VISUAL observations of phase transitions and other changes occurring in transparent solids and liquids subjected to extremely high pressures are now being made at the Bureau. To obtain these visual characterizations, Alvin Van Valkenburg of the crystallography laboratory has coupled a tiny diamond pressure cell [1] and a microscope which focuses through the diamond onto the specimen. Experiments at calculated pressures of 70 kilobars (1 million pounds) per square inch are being routinely undertaken and observations at pressures up to 115 kilobars have been made. To obtain detailed information on the phases present at the various pressures, an X-ray diffraction camera [2] which incorporates the diamond cell has been developed.

A study of the transitions that materials undergo at high pressures aids in defining interatomic forces and the perturbing effects of neighboring atoms on one another. By passing infrared (see footnote 1) and X-rays (see footnote 2) through the diamond pressure cell or viewing the transformations through a microscope, it is possible to relate changes in crystal structures to changes in bond energies, force constants, and vibration frequencies. The Bureau is studying these transformations and interactions at high pressures as part of its basic research program to determine intrinsic properties of matter at extremes of pressure and temperature.

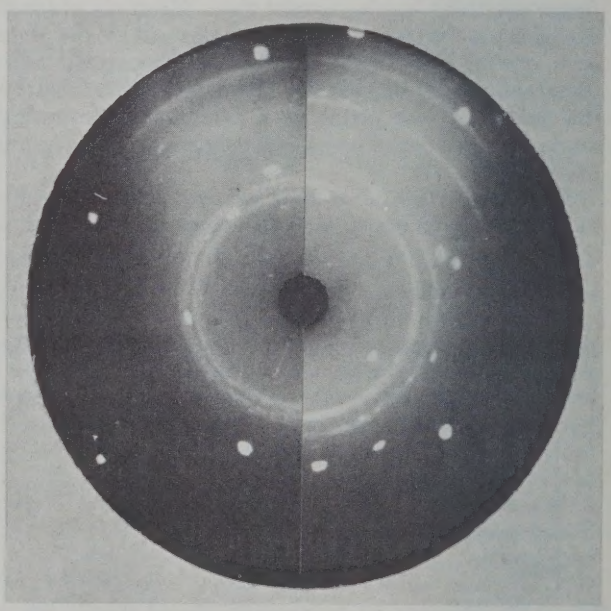
Although high-pressure diamond cells have been used for several years to study the infrared transmittance of substances, only recently have observations been carried out visually.

The anvil-type diamond pressure cell used in the experiments contains two carefully ground diamonds, each having a diameter of about $\frac{1}{16}$ in. For observing high-pressure transformations at room temperature, a specimen is placed between these two surfaces which are then squeezed together using a spring loading mechanism. This compresses the specimen into a film. Friction between the sample and diamond surfaces prevents the specimen from completely extruding. A polarized microscope with a special 16 magnification objective having a long working distance is then utilized to focus through one diamond onto the surface of the specimen. Thus far, thallium halides and halides of sodium, potassium, and silver, as well as a few other selected materials, have been studied by this procedure.

Transformations Observed

Because the pressure at the center of the diamonds is greater than at the periphery, any transition is first observed as a point. With increased pressure, this point spreads out into a ring. In cubic crystals, transitions can be observed as an island emerging at the center of the diamonds and then spreading out into a ring. The ring represents the boundary between the two polymorphic phases that have different indices of refraction.

Some of the visual effects accompanying phase transitions are extremely vivid. When potassium nitrate is viewed at atmospheric pressure with crossed nicols, many irregular but sharply defined brightly colored areas—which are the crystals of the compound—are seen. As pressure is gradually increased, these crystals grow and change shape rapidly, moving back and forth among themselves. At about 3.5 kilobars (45,000 lb/



Top of page: **Microphotograph of silver bromide crystals under 90 kilobars pressure (× 160) taken under polarized light. In cubic materials such as silver bromide, the circle represents the boundary between the two polymorphic phases that have different indices of refraction.** *Above:* **Matched X-ray powder diffraction patterns of thallium showing high- and low-pressure forms. Left half represents normal hexagonal thallium at 1 bar. Right half, taken at a calculated pressure of 60 kilobars, indicates a cubic structure. The powder diffraction pattern of the metal is indicated by the rings and arcs. The spots are due to diffraction by the diamonds.**

Studies—
d Visually

in.²), a phase transition occurs. These crystals produce a marbled field of variegated colors.

Teflon is equally impressive under a microscope. At atmospheric pressure this organic compound has a grainy, iridescent appearance. As pressure is increased just slightly (to about 100 lb/in.²), the crystal structure deforms suddenly into thousands of tiny swirls, which fade almost immediately into a mottled black background.

Crystal growth phenomena have been studied with the diamond squeezer and microscope. In potassium nitrate, for example, the crystals grown under high pressure are much smaller and denser than those propagated in the outer, lower pressure regions of the cell. The crystals in the low-pressure regions have a preferred orientation with their longest direction radial to the center; this is also the direction of the pressure gradient. By changing pressure, a growing crystal front that has the appearance of a liquid interface can be produced.

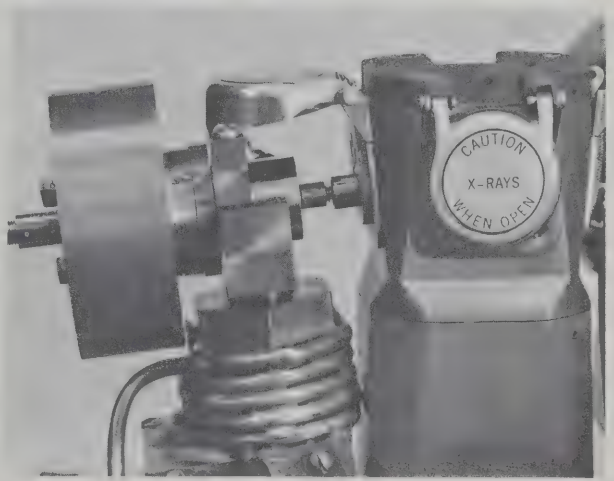
Absorption differences between two polymorphic phases cause another optical effect that may be used to detect the presence of two phases in cubic materials visually. When viewed in white or monochromatic light, the intensity difference observed between the high- and low-pressure forms of silver iodide is quite definite. The high-pressure phase has less intensity in the 0.4685-μ region than does the low-pressure form.

In birefracting materials, phase changes can be detected by observing the change in birefringence from a low- to a high-pressure phase or the elimination of birefringence on going from a noncubic to a cubic material. Hexagonal silver iodide is transformed from a noncubic to cubic phase under pressure. A definite boundary can be detected between these phases with or without crossed nicols, although the boundary effect appears to be more pronounced with the latter arrangement. Absorption changes indicate the transition between the phases.

An absorption phenomenon that has been observed in thallium halides does not appear to be related to a phase change. At calculated pressures of approximately 40 kilobars, thallium bromide changes from a near white to a lemon yellow color when observed in monochromatic light. As pressure is increased, this color changes to orange, then to a deep red, and finally at approximately 90 kilobars the material becomes opaque. On release of pressure the color reverts back to the original white. The same behavior is noted in thallium iodide and thallium chloride but at different pressures. These changes are related to the changes in optical absorption and electrical resistance reported by Drickamer [3] in the thallous halides at pressures up to 500 kilobars, and attributed by him to a decrease in the gap between the valence and conduction bands of

Alvin Van Valkenburg gradually applies pressure to the specimen in a diamond pressure cell while observing the phase transformations through a microscope.

X-ray diffraction camera which incorporates a diamond pressure cell. By passing X-rays through the diamond cell, structural information of the phase present at various pressures can be obtained.



allowed electronic energy. The color changes observed here are doubtless manifestations of the same shift in the optical absorption edge observed by Drickamer.

Liquids may also be visually examined with the diamond cell. In a recent experiment, conducted at room temperature, a 0.003-in.-thick platinum-foil gasket with a hole smaller than the diamond window was used to hold a drop of water between the windows. As pressure was applied, the gasket was squeezed into the opening containing the water, so that the water was compressed until it froze into a solid. By varying the pressure, at least two crystalline phases were detected. One such phase was growing into the liquid, whereas the second type was growing into the first.

X-ray Diffraction Apparatus

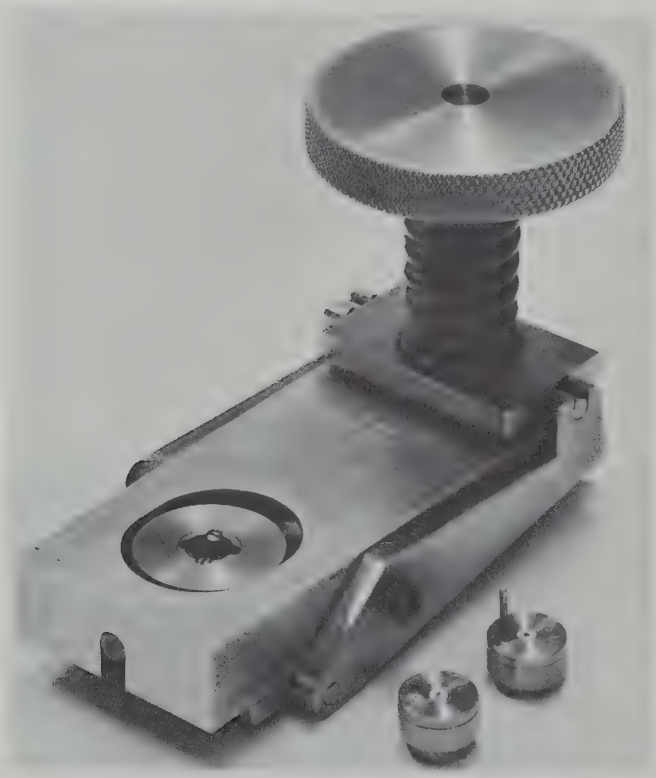
Adapting the diamond pressure cell for X-ray diffraction studies, C. E. Weir and G. J. Piermarini, also of the crystallography laboratory, have developed a high-pressure, X-ray diffraction powder camera. With this apparatus, X-ray beams, initiated from a molybdenum radiation source, are completely transmitted through the diamonds and the specimen. Diffraction rings from the compressed powdered specimen diverge through the diamond and are recorded on a film which is covered by a light-protective paper. The instrument is utilized in a routine manner at room temperature up to pressures of approximately 60 kilobars. In preliminary experiments undertaken so far, silver iodide, potassium bromide, bismuth, and thallium have been examined for high-pressure polymorphic transitions.

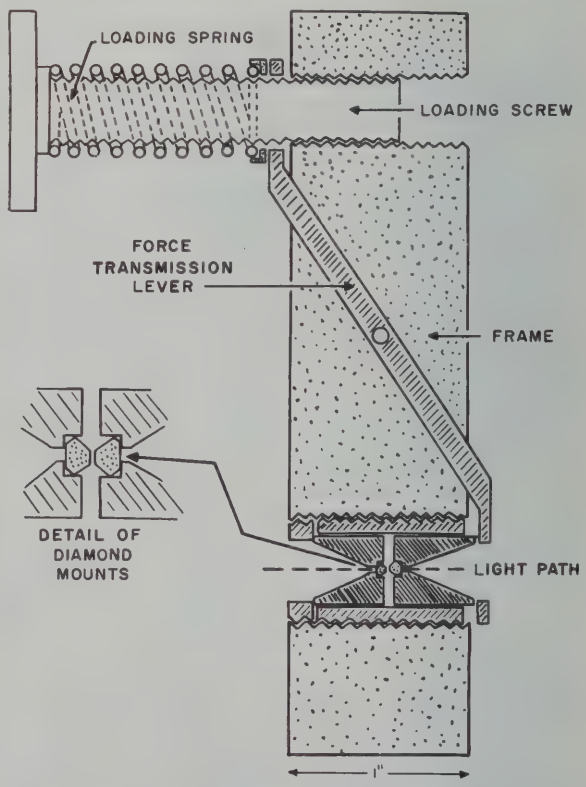
At present, the diamond pressure cell incorporated in the X-ray diffraction equipment differs slightly from the cell used for visual studies. Pressure for the former cell is applied by a piston actuated by a hydraulic liquid rather than by a spring loading mechanism. The pressure is measured with a bourdon-type gage. A valve is mounted in the liquid line between the pump and the gage so that the pressure cell and gage can be isolated from the pump during exposures. The dimensions of the unit and the diamond surface areas are such that the hydraulic pressure is magnified by a factor of approximately 500. Thus, to obtain a pressure of 60 kilobars on the specimen, a liquid pressure of approximately 2,000 psi is required.

[1] Infrared studies in the 1- to 15-micron region to 30,000 atmospheres, *C. E. Weir, E. R. Lippincott, A. Van Valkenburg, and E. N. Bunting, J. Research NBS* **63A** *(Phys. and Chem.), 55 (1959)*; High pressure effects on solids studied by infrared spectroscopy, *NBS Tech. News Bull.* **43,** *138 (1959)*.

[2] Diamond cell for X-ray diffraction studies at high pressures, *G. J. Piermarini and C. E. Weir, J. Research NBS* **66A4** *(Phys. & Chem.) 325 (July–Aug. 1962)*.

[3] The use of pressure to investigate the electronic structure of ionic crystals, *H. G. Drickamer, Proc. ONR Symp. Physics and Chemistry of Solids, at the Pennsylvania State University, May 1962 (in press, Gordon and Breech, N.Y.)*.





Left: **Diamond pressure cell used for applying high pressure to solids. With this apparatus, pressures up to 115 kilobars can be obtained. As the spring** *(top right)* **is tightened, a lever system transmits pressure to two diamond holders** *(lower right)* **which are inserted in the circular opening at the bottom. When in use, the flat sides of the holders face one another and very small diamonds are held at the center of each holder. The specimen is compressed between the diamond surfaces.** *Right:* **Schematic diagram of "squeezer."**

Amine Buffers for *p*H Control

FOR THE ACCURATE control of acidity and basicity in the *p*H range 7 to 9—the area most vital to biological and medical research—the Bureau is developing and studying a series of organic buffer solutions called ammonium or amine buffers. The physicochemical and thermodynamic properties of these buffers are being determined. Studies of these compounds are an important first step toward procedures for the regulation of acidity at a desired *p*H.[1, 2]

In general, a buffer solution consists of a weak acid and its salt or a weak base and its salt. Such solutions resist changes in hydrogen-ion concentration when a small amount of an acid or base is introduced. That is, the buffer stabilizes the system so that the *p*H, i.e., the degree of acidity or basicity, remains virtually unchanged, even though acidic or basic contaminants may be added.

The investigation of amine buffers is part of a continuing research program which R. G. Bates and coworkers of the solution chemistry group are conducting to determine thermodynamic properties of electrolytic solutions. Demands have increased for expanding basic knowledge and the usefulness of routine electrometric measurements of solutions containing solvents other than water. NBS studies of the thermodynamic constants of acids and bases and of acid-base interactions in solutions provide data essential to the development, improvement, and extension of methods for measurement and control of acidity.

Because very few weak acids with ionization constants between 10^{-7} and 10^{-9} (i.e., *p*H 7 to 9) are known, suitable primary standards for titrating strong acids are difficult to select. Salts of the very weak acids are likely to be unstable in the presence of moisture and carbon dioxide, whereas those of the stronger acids are not completely neutralized except at *p*H values too low to obtain sharp end points. Consequently, acid-salt buffer systems suitable for the regulation of *p*H in this physiologically important *p*H range are not numerous. Furthermore, most of the available buffer solutions are incompatible in one way or another with physiological media.

The NBS scientists have found that some of these difficulties can be overcome by using organic nitrogen bases as buffers. For effectively utilizing the organic nitrogen bases in titrations and in the regulation of acidity, knowledge of the ionization constant, preferably over a range of temperatures, is necessary. Although careful measurement of the ionization constant exponent, or *p*K values, of weak acids and related thermodynamic constants has received much attention in the past three decades, such values for the ammonia bases have been largely neglected.[3] These substances were not studied because of (1) the difficulty of preventing part of the volatile base from being removed by the bubbling hydrogen—which is necessary for proper functioning of the hydrogen electrode—and (2) the appreciable solubility of the silver-chloride electrode in many solutions of nitrogen bases.

However, results of a study on ammonia indicated that previous difficulties could be eliminated with improved experimental design and suitable corrections to the data.[4] Procedures incorporating these changes have been applied to the investigation of 4-aminopyridine,[5] tris(hydroxymethyl)aminomethane,[6] and the dihydroxy analog of the latter, namely, 2-amino-2-methyl-1,3-propanediol.[7] In addition, piperidine[8] and mono-, di-, and triethanolamine[9, 10, 11] have been investigated.

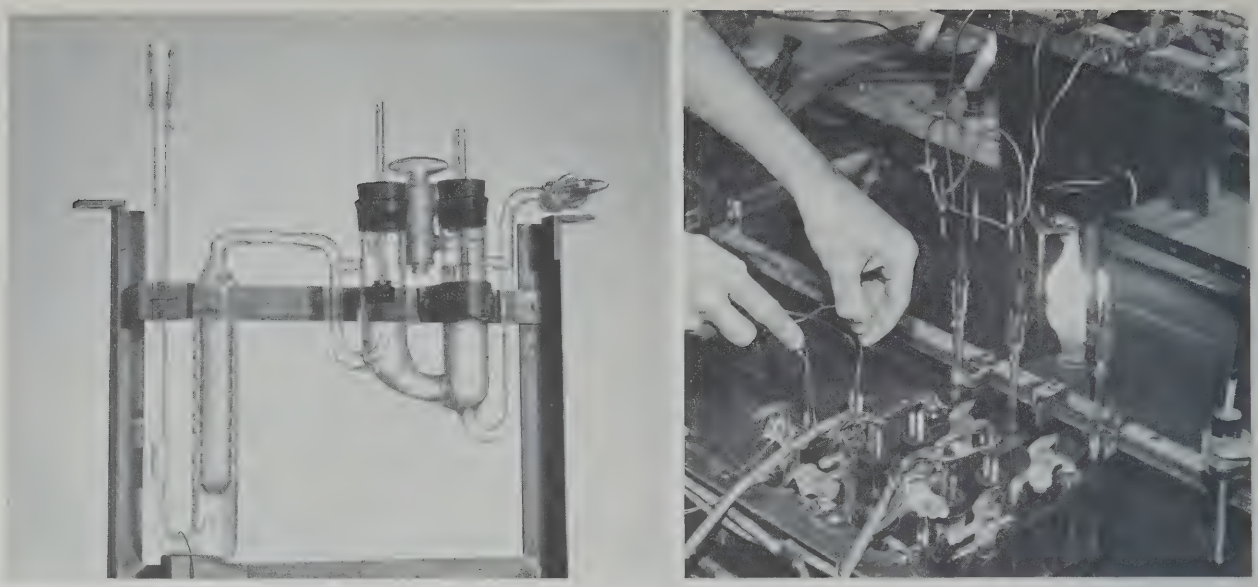
The ammonium ion has a *p*K of 9.24 at 25 °C. The substitution of aliphatic organic radicals such as methyl or ethyl is known to weaken the acidic strength of ammonium ion, but groups which contain the electron-attracting hydroxyl (OH) group increase the strength of ammonium ion as an acid. Thus it is possible to select the proper substituent to produce an acid of approximately the desired strength. The resulting ammonia bases are compatible with calcium salts and with most physiological fluids.

Many of the organic-substituted ammonia bases are liquids that are difficult to purify and have limited stability in air. However, in the present study two solid amines were found to be very suitable as acidimetric standards for regulating *p*H in the intermediate range. These amines—4-aminopyridine and *tris*(hydroxymethyl)aminomethane, $(CH_2OH)_3CNH_2$—are stable, crystalline substances which can be obtained commercially in a high degree of purity. They are strong enough bases to be completely neutralized by strong acids at *p*H values of 4.8 and 5.3 respectively, in 0.05 *M* solution. Because of their solubility in many nonaqueous media as well as in water, they may later be utilized in the standardization of nonaqueous acidimetric titrations. Suitable standards capable of bridging the gap between aqueous and nonaqueous solutions are difficult to find.

Another factor that makes these two substances suitable for the regulation of *p*H is the relatively low pK values for their protonated forms (9.11 and 8.07, respectively). In fact, the weakly basic character of *tris*(hydroxymethyl)aminomethane, coupled with its compatibility with the buffer systems of the blood stream, has led to the *in vivo* administration of this base as a clinical aid in the correction of respiratory acidosis.

Experimental Procedure

The NBS method of determining ionization constants of the substituted amines is based on electromotive force measurements. It is a modification of a procedure developed by Harned and coworkers at Yale University in the early 1930's. From the measurements, *p*K results which are precise to within 0.002 unit have been obtained when the weakly acidic species has a *p*K between 2.5 and 11.5. The cells utilize two



Typical cell (*left*) used for measuring electromotive force of amine buffers. The cell utilizes two types of electrodes: silver-silver chloride and hydrogen gas. These electrodes are separated by a stopcock to prevent diffusion of dissolved silver into the hydrogen electrode. For actual measurements, the cells are placed in a constant-temperature bath (*right*) to avoid temperature changes.

types of electrodes which are among the most reproducible known. These are the silver-silver chloride electrode and the hydrogen-gas electrode, which is the primary standard for all measurements of acidity. The standard reference potential of the silver-silver chloride electrode, which is the basis of considerable electrochemical data, has been redetermined at the Bureau [12] over a wide temperature range.

For studying volatile acids and bases, the method was modified by (1) inserting a stopcock between the electrode compartments to prevent diffusion of dissolved silver to the hydrogen electrode, and (2) applying a correction factor for the solubility of silver chloride in solutions of the nitrogen bases.

For determining the ionization constant, cells containing the free amine and its hydrochloride in aqueous solution are studied at eleven temperatures from 0 to 50 °C over a period of 3 days. The electromotive force is reproducible to better than 0.0001 v (that is, 0.0017 *p*K unit) and is usually stable within this tolerance over the three days required to complete the measurements. Careful temperature control is particularly important in the study of amine bases, because the *p*H of most amine buffer solutions falls by about 0.03 unit for every increase of 1 deg C in the temperature.

Effect of Structural Changes

The substitution of hydroxyl groups into the molecule of an ammonia base lowers not only the volatility of the base but also the basic strength. Ammonia is a gas at ordinary temperatures, but the ethanolamines are liquids with boiling points that rise progressively from monoethanolamine to triethanolamine. Analogously, the *p*K values have been found to drop from 9.50 for monoethanolammonium ion to 7.76 for triethanolammonium, whereas replacement of the methyl hydrogens of methylammonium by hydroxymethyl groups lowers the *p*K from 10.62 progressively to 9.69, 8.79, and finally to 8.08 when three hydroxymethyl groups have been introduced.

The high accuracy of the *p*K values has made it possible to derive from the measurements the changes of heat content, entropy, and heat capacity accompanying the ionization of the base under study. These quantities are of practical as well as theoretical interest. For example, the change of heat content indicates the effect of temperature upon the *p*H of buffer solutions composed of the base and its salt, whereas the changes of entropy and heat capacity and their correlation with the structure of the base shed light on the mechanism of the ionization process. The nitrogen bases are particularly well suited for this purpose, inasmuch as the ionization of a cation acid of the ammonium type is an isoelectric process. That is, the ionization takes place without the creation of an electric charge—the positive charge on a substituted ammonium ion disappears and a hydrogen ion appears in its place. Thus, no electrostatic contribution to the entropy and heat capacity is present to obscure changes associated with alterations in the structure of the bases.

The measurements of entropy and heat capacity for the dissociation of bases appear to support the theory that orientation of water molecules around the acid cation and basic molecule plays a significant part in the ionization process. Bulky groups attached to the

nitrogen atoms tend to exclude solvent and to reduce the entropy of hydration of the ion. The well-known hydrophobic character of alkyl groups, that is, their very weak affinity for water, appears to produce a similar effect. The opposite effect is observed when the alkyl chain is lengthened. Analogously, the addition of hydroxyl groups to the base molecule results in a larger negative entropy of ionization, presumably because these groups are water-attracting. Thus, from the Bureau data, useful predictions can be made regarding the effect of substituents on the strength of the base.

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[2] Amine buffers for pH control, *R. G. Bates, Ann. N.Y. Acad. Sci.* **92,** *340 (1961).*

[3] *See, for example, R. A. Robinson and R. H. Stokes,* Electrolyte Solutions, *2d ed., appendix 12.1, Academic Press, Inc., New York, N.Y. (1959).*

[4] The acidic dissociation constant of ammonium ion at 0 to 50 °C, and the base strength of ammonia, *R. G. Bates and G. D. Pinching, J. Research NBS* **42,** *419 (1949).*

[5] Dissociation constant of 4-aminopyridinium ion in water from 0 to 50 °C and related thermodynamic quantities, *R. G. Bates and H. B. Hetzer, J. Research NBS* **64A** *(Phys. and Chem.), 427 (1960).*

[6] Dissociation constant of the protonated acid form of 2-amino-2-(hydroxymethyl)-1,3-propanediol [tris-(hydroxymethyl-aminomethane] and related thermodynamic quantities from 0 to 50°, *R. G. Bates and H. B. Hetzer, J. Phys. Chem.* **65,** *667 (1961).*

[7] Dissociation constant of 2-ammonium-2-methyl-1,3-propanediol in water from 0 to 50° and related thermodynamic quantities, *H. B. Hetzer and R. G. Bates, J. Phys. Chem.* **66,** *308 (1962).*

[8] Dissociation constant for piperidinium ion from 0 to 50 °C and related thermodynamic quantities, *R. G. Bates and V. E. Bower, J. Research NBS* **57,** *153 (1956).*

[9] Acidic dissociation constant and related thermodynamic quantities for monoethanolammonium ion in water from 0 to 50 °C, *R. G. Bates and G. D. Pinching, J. Research NBS* **46,** *349 (1951).*

[10] Acidic dissociation constant and related thermodynamic quantities for diethanolammonium ion in water from 0 to 50 °C, *V. E. Bower, R. A. Robinson, and R. G. Bates, J. Research NBS* **66A** *(Phys. and Chem.), 71 (1962).*

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[12] Standard potentials of the silver-silver-chloride electrode from 0 to 95 °C and the thermodynamic properties of dilute hydrochloric acid solutions, *R. G. Bates and V. E. Bower, J. Research NBS* **53,** 283 *(1954).*

Graduate Work-Study Program Initiated

THE BUREAU recently instituted a unique Government work-study program which will allow graduate students in the Washington area to complete courses for advanced degrees in science at approximately two-thirds of the average pace while earning 70 percent of a junior scientist's salary.

The graduate work-study plan, which was designed by the Bureau in cooperation with local universities, will begin in September. Students may work in an NBS lab 3 days a week while taking courses on a university campus each Tuesday, Thursday, and Saturday. Although the plan will vary to meet requirements of a specific university, it is expected that the Ph. D. program can generally be completed in 5 years. After finishing course work in 4 years, the participants will have the option of completing thesis research at the Bureau or at the university. Other research laboratories in the area are also expected to participate in work-study programs at the graduate level.

Catholic University already has part-time course programs available at the graduate level in physics, chemistry, and mechanical engineering. The University of Maryland has a suitable schedule in the graduate physics curriculum. Other universities may announce similar programs in the future.

Students will enter the Bureau at a GS–7 level. With both satisfactory job performance and academic progress, they may be promoted to a GS–11 level while in training. They will work full time at NBS during the summer.

Aside from its financial advantages, the program offers an excellent opportunity to gain practical laboratory experience. Work opportunities are available in the Bureau's technical divisions for physicists, chemists, mathmaticians, mechanical engineers, and electrical engineers with a B.S. degree. Students will work in their chosen field of interest.

The payment of tuition and fees will be the personal responsibility of the partcipants with no eligibility for reimbursement by the Bureau. There is no continued service obligation. Interested scientists should first be admitted to a graduate school in the Washington area, and then contact:

Mrs. Ruth B. Armsby,
Employee Development Officer
Personnel Division
National Bureau of Standards
Washington 25, D.C.

Hollomon Appointed Assistant Secretary for Science and Technology

Dr. John Herbert Hollomon, former General Electric scientist-engineer and administrator, was administered the oath of office on Monday, May 14, 1962, as the first Assistant Secretary of Commerce for Science and Technology by Secretary of Commerce Luther H. Hodges.

In this new post, created by Congress on February 17, 1962, Dr. Hollomon will direct the scientific and technical functions of the Department and will be the principal adviser to the Secretary on all scientific and technological matters of concern to the Department.

Dr. Hollomon recently outlined his duties in an address to the 47th National Conference on Weights and Measures. The Assistant Secretary said that he had three main jobs. The first would be to bring to Secretary Hodges' attention the importance of science, technology, research and development, invention, and standards so that the Secretary might better be able to serve the President.

His second responsibility would be that of helping to establish policy for the technical agencies of the Department, including the National Bureau of Standards, the Weather Bureau, the Coast and Geodetic Survey, and the Patent Office. Dr. Hollomon is also a member of the Federal Council for Science and Technology, which is chaired by Dr. Jerome Weisner. The FCST sets policy for the entire Federal research and development program, and, through Dr. Weisner, advises the President.

Dr. Hollomon said one of his prime goals is to help put research and development to more effective use in promoting the Nation's economic growth. He also pointed out that the success of the Department's mission to expand international commerce will depend not only on economic factors, such as tariffs and monetary policies, but very significantly on the more effective cooperation among the nations of the world in developing common standards of weights and measures.

Dr. Hollomon is a native of Norfolk, Va., where he was born March 12, 1919. He received his BS and Doctor of Science degrees at M.I.T. and taught a year at Harvard. Dr. Hollomon has been associated with the General Electric Company since 1946, having joined its research laboratory as Assistant Manager of the Metallurgy Research Department. In 1960 he was appointed General Manager of the General Engineering Laboratory of the company.

Dr. Hollomon has been an Adjunct Professor of Metallurgy at Rensselaer Polytechnic Institute and has served in advisory educational posts at Cornell, Harvard, and the Massachusetts Institute of Technology. He is the author of a textbook on metallurgy and has written or co-authored more than 50 articles in professional journals.

47th National Conference on Weights and Measures

Standardization of sizes of prepackaged commodities was presented as a nationwide problem and was the main topic of discussion during the sessions of the 47th National Conference on Weights and Measures held in Washington, D.C., June 4 to 8, 1962.

More than 400 delegates heard Senator Philip A. Hart of Michigan (Chairman) of the Senate Antitrust and Monopoly Subcommittee, describe the need for a Federal statute that would require certain standardization of package sizes and labels, thus making it possible for consumers to make easy and direct comparisons of the quantities of contents of packages. The adoption of definite standards would insure that the information necessary for making a rational purchase would be plainly located on every package, expressed in terms that are meaningful, readily understood, and easily transferable for value comparisons.

Others who spoke on the subject of packaging were F. J. Schlink, President and Technical Director of Consumers' Research in Washington, N.J., who described some of the ways consumers are being deceived through slack filling of packages, and E. A. Vadelund, Assistant Director of the Pennsylvania Bureau of Standard Weights and Measures, who reported on the package control plan now in use in his State.

Dr. J. Herbert Hollomon, Assistant Secretary of Commerce for Science and Technology, spoke of the need for international cooperation in standards and commerce during his keynote address; Dr. A. V. Astin, Director of the National Bureau of Standards and President of the Conference, discussed the weights and measures program of the National Bureau of Standards and recent accomplishments of the Bureau and presented Honor Awards; and Siguard Anderson, Commissioner of the Federal Trade Commission, addressed guests at the Conference luncheon, relating some of the deceptive practices which the FTC has uncovered. Weights and measures officials from abroad who presented papers included Mitsuo Tamano, Director of the National Research Laboratory of Metrology of Japan, who discussed the transition to the metric system of measurement in his nation; Thomas Cairns, Chief Inspector of the Weights and Measures Department in Glasgow, Scotland, who presented a paper on weights and measures in Scotland, ancient and modern; and R. W. MacLean, Director, Standards Branch, Department of Trade and Commerce of Canada, who described recent experiences of his nation in the area of pattern approval of commercial weighing and measuring devices.

The National Conference on Weights and Measures, which was organized in 1905, is sponsored by NBS. This year the Conference brought together weights and measures officials representing 38 States, the District of Columbia, and the Commonwealth of Puerto Rico, as well as representatives of business and industry. Model laws, specifications, tolerances, regulations, and enforcement practices are recommended by the Conference for adoption by various States, which have the legal responsibility of regulating commercial weighing and measuring devices and of controlling commercial transactions involving quantity.

The Bureau, through its Office of Weights and Measures, cooperates with the States in their endeavor by providing reference standards, calibrating services, and a wide range of technical advisory programs. Thus, the national standards of length and mass, in the custody of the Bureau, are translated into everyday use.

Officers of the Conference elected for the coming year include *Chairman:* C. H. Stender (South Carolina); *Vice Chairmen:* C. L. Jackson (Wisconsin), P. I. Morris, Jr. (Georgia), Nathan Kalechman (Hartford, Connecticut), and J. F. McCarthy (Boston, Massachusetts); *Treasurer:* C. C. Morgan (Gary, Indiana); and *Chaplain:* R. W. Searles (Medina, Ohio). A. V. Astin will continue as *ex officio* President of the Conference, and W. S. Bussey, Assistant to the Director for Weights and Measures Administration, will continue as *ex officio* Executive Secretary.

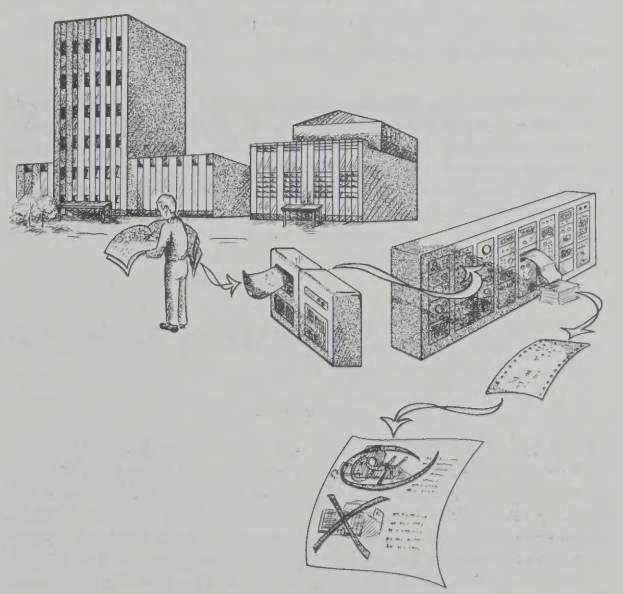
NBS Cooperates in Fallout Shelter Evaluation Program

THE BUREAU is participating in an Office of Civil Defense program to assess the value of large buildings as fallout shelters. This nationwide survey will identify not only those buildings or parts of buildings that can serve as fallout shelters as they stand, but also those which, through some modification, can be rendered suitable for this purpose. The survey, which is expected to be completed some time this year, will help identify those geographic areas in which sufficient shelter space exists, as well as those in which there is a need for more shelter. Other Government agencies assisting in the survey are the Census Bureau, the Army Corps of Engineers, and the Navy Bureau of Yards and Docks.

NBS involvement in the program is twofold. The Bureau's radiation theory laboratory, under the direction of Lewis V. Spencer, developed the mathematical procedures and theoretical data necessary for estimating the protection factors [1] of a wide variety of buildings.[2] These procedures are being used by the computation laboratory, headed by D. I. Mittleman, to convert field data on actual structures to protection factors. Such a large-scale program was considered appropriate, in terms of economics or time, only with the use of high-speed electronic data processing.

Data on an estimated several hundred thousand buildings across the nation, covering such variables as room size, wall composition and thickness, number of floors, and others, are being collected by qualified architects and engineers. The detailed information on each building is entered on a large standard form, developed by the Census Bureau in close cooperation with the Office of Civil Defense, Department of Defense, and the Corps of Engineers, U.S. Army. The completed form for each building is sent to the Census Bureau where it is microfilmed and then, with FOSDIC (film optical sensing device for input to computers),[3] the data are transferred to a magnetic tape for input to the NBS computer.

At NBS the data are first edited for errors and consistency, and then processed by a computer, a protection factor being derived for each story of each building. Modifications of the input program have reduced the computational time for each story of a building from the original value of 4 sec to about 0.05 sec.



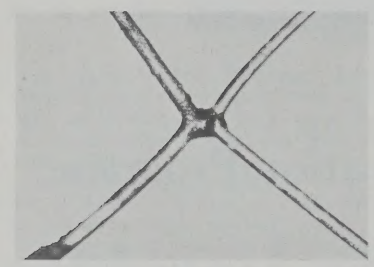
Artist's conception of the current nationwide program to determine the suitability of existing structures as fallout shelters. The Bureau is carrying out the mathematical and computational aspects of the study under Office of Civil Defense sponsorship. Data on individual buildings, collected in the field by architects and engineers, are recorded on magnetic tape by FOSDIC, then processed at NBS on high-speed computers. Protection factors are derived, indicating whether or not a particular building will provide adequate shielding from fallout radiation.

The completed data for each building within a geographic district are printed out as a unit. It then becomes the task of the Army Corps of Engineers or the Navy Bureau of Yards and Docks, depending on which has responsibility in a certain area, to identify usable shelters. As the program progresses, local Civil Defense Offices will provide each shelter with supplies sufficient to sustain the number of persons sheltered for a specified time.

[1] *The protection factor is defined as the ratio of the detector response in a standard position outside of the building to that within the building.*

[2] Structure shielding against fallout radiation from nuclear weapons, *NBS Mono. 42, available from the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C., 75 cents. See also* Structure shielding against fallout radiation, *NBS Tech. News Bull.* **46,** *73 (June 1962).*

[3] FOSDIC III to assist in 1960 census, *NBS Tech. News Bull.* **43,** *106 (June 1959).*



Microsize Magnetic Field Probes

MICROSIZE magnetic field probes—occupying a volume of less than 10^{-8} ml—have been devised by Carl A. Shiffman of the cryogenic physics laboratory. The probes, which were developed in a project sponsored by the Air Force, utilize the magnetoresistance effect and can be made with axially symmetric sensitivity.[1] They are intended for use at liquid nitrogen temperatures and below.

The small size of probes of this type permits them to be placed in close proximity to the surface of the material being studied. Shiffman has achieved a separation of only 5 μ in an investigation of the intermediate state of long cylindrical semiconductors. In addition, the small size and axial symmetry make accurate positioning and orientation unnecessary. The fact that they are operable at very low temperatures suggests their use in the investigation of superconducting solenoids of very high field.

Above: **Microsize magnetic field probe. The four leads consist of copper-gold wire 20 μ in diameter. The probe (short cylinder joining the V-shaped leads) consists of a single crystal of bismuth occupying a volume of less than 10^{-8} ml, and is for use at the temperature of liquid nitrogen and below.**

The probes are easily formed by dipping a V-shaped copper-gold (50 percent each) wire of 20 μ diam into molten bismuth and drawing out a miniature ball. This procedure always results in the formation of a single crystal of bismuth. Another V is attached to the free end of the crystal to form a four-lead network.

The anisotropy of the crystal lattice is dominant in determining the symmetry of the magnetoresistance. Usually, the crystals grow with the 3-axis normal to the cylinder axis, resulting in rather infrequent production of axial symmetry. This defect can be remedied by recrystallizing the bismuth. To accomplish this, the probe is heated from below with a soldering iron. The position of the iron can be adjusted so that cooling causes the crystal to solidify with the liquid-solid interface traveling almost vertically. This procedure produces probes having the desired symmetry about half the time.

Mounts for the completed probes are made from small brass sheets coated with an epoxy resin having a coefficient of expansion similar to that of the brass. Copper leads are imbedded in the resin, and the leads of the probe are bonded to the mount with the same resin. After polymerization of the resin, the ends of the V's are soldered to the copper wires, and the probe is ready for use.

[1] *For further details, see* Microsize magnetic field probes with axial symmetry, *by Carl A. Shiffman, Rev. Sci. Instr.* **33,** *206 (Feb. 1962).*

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Journal of Research of the National Bureau of Standards

Section A. Physics and Chemistry. Issued six times a year. Annual subscription: Domestic, $4; foreign, $4.75.

Section B. Mathematics and Mathematical Physics. Issued quarterly. Annual subscription: Domestic, $2.25; foreign, $2.75.

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Section D. Radio Propagation. Issued six times a year. Annual subscription: Domestic, $4; foreign, $4.75.

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Patents

The following U.S. Patents have recently been granted on NBS inventions and, except as noted, are assigned to the United States of America as represented by the Secretary of Commerce:

3,009,121. Nov. 14, 1961. Adjustable Frequency Rejection Filter. William V. Loebenstein (licensed to the United States of America as represented by the Secretary of Commerce).

3,025,000. Mar. 13, 1962. Function Generator for Generating a Function of Two Independent Variables. Leonard Taback.

3,028,351. Apr. 3, 1962. Pressure Sensitive Adhesive Composition, Comprising Polyvinylpyrrolidone and Polyethylene Polyamine, Article Coated Therewith and Method of Making Same. Karl F. Plitt.

3,028,755. Apr. 10, 1962. Apparatus for Water Penetration Testing of Sole Leather. Thomas J. Carter.

3,030,577. Apr. 17, 1962. Apparatus for Calibrating Microwave Reflectivity Coefficient Standards. Alan C. Macpherson.

3,030,806. Apr. 24, 1962. Seebeck-Peltier Flowmeter. Merlin Davis.

3,031,617. Apr. 24, 1962. Linear Capacitive Probe Detecting Device. Donald R. Paquette.

3,031,914. May 1, 1962. Parallel Testing Interferometer. James B. Saunders.

3,032,611. May 1, 1962. Combined Frequency-Phase Modulation Telegraph System. George Franklin Montgomery (Licensed to the United States of America as represented by the Secretary of Commerce).

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3,034,332. May 15, 1962. Step Function Pressure Calibrator. Paul S. Lederer (NAVY).

3,034,397. May 15, 1962. Parallel Testing Interferometer. James B. Saunders.

3,036,222. May 22, 1962. Plug-in Packages for Electronic Circuits. Richard P. Witt.

3,041,533. June 26, 1962. RF Voltmeter Calibration Console. Myron C. Selby, Lewis F. Behrent, and Francis X. Ries.

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